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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

JAN 27 1994

OFFICE OF
PREVENTION, PESTICIDES AND
TOXIC SUBSTANCES

MEMORANDUM

SUBJECT: **Mepiquat Chloride.** Magnitude of the Residue in Cottonseed and Cottonseed Processed Commodities. Reregistration Case No. 2375. Chemical No. 109101. MRID #42734601 and 42734602. DP Barcode D192305. CBRS #12,046.

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The Mepiquat Chloride Phase 4 Review (S. Funk, 1/15/91) specified data requirements for cotton forage and cottonseed, and for cotton processed commodities. In response, BASF Corporation (1992; MRIDs 42426802, 42426803, and 42554111) has submitted residue data for cottonseed and cottonseed processed commodities which have been reviewed (J.Stokes, 10/12/93, CBTS #10671, 106890, and 10690). BASF Corporation (1993; MRIDs 42734601 and 42734602) has also submitted additional magnitude of the residue data for cottonseed, reflecting ULV and multiple low-rate applications, in response to Phase 4 requirements and to support proposed label amendments. These data have been reviewed by Acurex under contract to the Agency, and have undergone secondary review to reflect Branch policies.

Data in this submission and previously submitted data (see J.Stokes, 10/12/93, CBTS #10671, 106890, and 10690) indicate that method A9106 is adequate for collecting data on

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residues of mepiquat chloride per se in or on cottonseed, provided that the registrant adequately clarifies whether in the current submission cottonseed samples were homogenized before extraction. If whole cottonseeds, rather than cottonseed homogenate, were extracted, the registrant must provide comparative data showing the extraction efficiency for mepiquat chloride residues from samples bearing measurable, weathered residues and extracted both with and without homogenizing as part of the extraction process.

The submitted summary storage stability data indicate that residues of mepiquat chloride per se are stable in or on frozen cottonseed for the storage intervals reflected in the current submission.

The submitted data adequately fulfill outstanding magnitude of residue data requirements for cottonseed, and adequately support the registrant's proposed label amendments, provided that the registrant adequately addresses concerns regarding the extraction of samples for analysis. Residues were $\leq$ 1.24 ppm in or on cottonseed harvested 30 days following the last of up to 6 applications at 0.011-0.044 lb ai/A/application (total 0.132 lb ai/A; 2x the current maximum labeled rate). These data indicate that residues in or on cottonseed are not likely to exceed the current tolerance of 2 ppm following treatment at the rates tested.

Data requirements remain outstanding for cotton forage.

If you need additional input, please advise.

Attachment.

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MEPIQUAT CHLORIDE
(Chemical Code 109101)
(CBRS No. 12046; DP Barcode D192305)

TASK 2B

Phase 5-
Reregistration Review
Residue Chemistry

September 20, 1993

Contract No. 68-DO-0142

Submitted to:

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MEPIQUAT CHLORIDE

(Chemical Code 109101)

(CBRS No. 12046; DP Barcode D192305)

PHASE 5 - REREGISTRATION REVIEW

RESIDUE CHEMISTRY

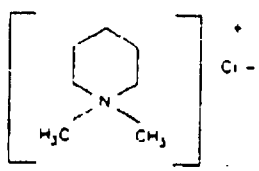
Task 2B

BACKGROUND

The Mepiquat Chloride Phase 4 Review (S. Funk, 1/15/91) specified data requirements for cotton forage and cottonseed, and for cotton processed commodities. In response, BASF Corporation (1992; MRIDs 42426802, 42426803, and 42554111) has submitted residue data for cottonseed and cottonseed processed commodities; these data have been reviewed by CBTS (J.Stokes, 10/12/93, CBTS #10671, 106890, and 10590). BASF Corporation (1993; MRIDs 42734601 and 42734602) has also submitted additional magnitude of the residue data for cottonseed, reflecting ULV and multiple low-rate applications, in response to Phase 4 requirements and to support proposed label amendments. These data are reviewed here to determine their adequacy in fulfilling outstanding data requirements and in supporting the proposed label amendments. The Conclusions and Recommendations stated below apply only to the magnitude of the residue in or on cottonseeds.

The qualitative nature of residue of mepiquat chloride in plants is adequately understood. The parent compound, mepiquat chloride, is the residue of concern. Method I in PAM, Vol. II, is described as very specialized and having recoveries in the 50% range, but is considered adequate for tolerance enforcement purposes. Tolerances for residues of mepiquat chloride are currently expressed in terms of N,N-dimethylpiperidinium chloride [40 CFR §180.384, §185.2275(a), (b), and (c), and §186.2275(a) and (b)].

There are no established or proposed Codex MRLs for mepiquat chloride. Therefore, no compatibility questions exist with respect to U.S. tolerances and Codex.



Mepiquat chloride

CONCLUSIONS

1. Data in this submission and previously submitted data (see J.Stokes, 10/12/93, CBTS #10671, 106890, and 10690) indicate that method A9106 is adequate for collecting data on residues of mepiquat chloride per se in or on cottonseed, provided that the registrant adequately clarifies whether cottonseed samples were homogenized before extraction. If whole cottonseeds, rather than cottonseed homogenate, were extracted, the registrant must provide comparative data showing the extraction efficiency for mepiquat chloride residues from samples bearing measurable, weathered residues and extracted both with and without homogenizing as part of the extraction process.

2. The submitted summary storage stability data indicate that residues of mepiquat chloride per se are stable in or on frozen cottonseed for the storage intervals reflected in the current submission.

3a. The submitted data adequately fulfill outstanding magnitude of residue data requirements for cottonseed, and adequately support the registrant's proposed label amendments, provided that the registrant adequately addresses concerns regarding the extraction of samples for analysis. Residues were ≤ 1.24 ppm in or on cottonseed harvested 30 days following the last of up to 6 applications at 0.011-0.044 lb ai/A/application (total 0.132 lb ai/A; 2x the current maximum labeled rate). These data indicate that residues in or on cottonseed are not likely to exceed the current tolerance of 2 ppm following treatment at the rates tested.

3b. Data requirements remain outstanding for cotton forage.

DETAILED CONSIDERATIONS

Residue Analytical Methods

BASF Corporation (1993; MRIDs 42734601 and 42734602) submitted residue data for mepiquat chloride collected from cottonseeds using the ion chromatography method A9106. The registrant stated that a complete report of method A9106 was submitted in MRID 42426801. This method has been reviewed by CBTS (J.Stokes, 10/12/93, CBTS #10671, 106890, and 10690) and found to be adequate for the determination of mepiquat chloride residues in/on cottonseed. In the current submission, the method was summarized and a flowchart of the method was presented, along with representative chromatograms.

Briefly, samples are extracted with 25% 0.5N HCl in methanol, then made basic and filtered. The filtrate is washed with hexane and dichloromethane, and the active ingredient is isolated as a dipicrylamine complex, which is partitioned into dichloromethane, decomplexed and extracted with HCl. Following an additional purification using alumina column chromatography, residues are quantified by ion pair chromatography with conductivity

detection using a suppressor system. The registrant stated that the method limit of quantitation is 0.1 ppm.

Method validation data were not included in the current submission. Concurrent method recoveries are presented in Table 1. Apparent residues of mepiquat chloride in or on cottonseed control samples were all <0.1 ppm; however, concurrent method recoveries were corrected for extrapolated apparent residues <0.1 in or on control samples.

Table 1. Concurrent method recoveries from cottonseed using method A9106.

MRID	Fortification Level (ppm)	No. of Samples	% Recovery
42734601	0.1	2	65, 93
	0.5	4	65-89
	2	3	71-81
	5	1	68
42734602	0.1	4	72-92
	0.5	7	66-89
	2	5	69-94

The registrant stated in the summary text of the current submissions that whole cottonseed samples, rather than homogenized cottonseeds, were extracted. The method flowchart calls for homogenization as the first step of extraction, and the method modifications do not indicate that this step was omitted. The registrant must clarify this statement.

The submitted data indicate that method A9106 is adequate for collecting data on residues of mepiquat chloride per se in or on cottonseed, provided that the registrant adequately clarifies whether cottonseed samples were homogenized for extraction. If whole cottonseeds, rather than cottonseed homogenate, were extracted, the registrant must provide comparative data showing the extraction efficiency for mepiquat chloride residues from samples bearing measurable, weathered residues and extracted both with and without homogenizing as part of the extraction process.

Storage Stability Data

BASF Corporation (1993; MRIDs 42734601 and 42734602) submitted summary storage stability data for weathered residues of mepiquat chloride per se in or on cottonseed, cottonseed hulls, and cottonseed meal stored frozen for up to 11 months after initial analyses (Table 2). The summary data indicate that residues of mepiquat chloride were stable in or on all matrices for the duration of the study. Treated cottonseed samples in the current submissions were stored frozen (≤ -5 °C) for up to 185 days (approximately 6 months) from time of harvest until extraction for analysis.

The submitted summary storage stability data indicate that residues of mepiquat chloride per se are stable in or on frozen cottonseed for the storage intervals reflected in the current submission.

Table 2. Summary storage stability data of weathered residues of mepiquat chloride per se found at initial analysis and after frozen storage interval (months).

Matrix	Uncorrected residues (ppm)[a] at initial analysis and after frozen storage (months)			
	0	5	10.5	11
Delinted seed	2.87 (84)[b]	--	--	2.62 (84)
Hulls	0.61 (88)	0.55 (69)	0.60 (77)	--
Meal	5.0 (90)	4.9 (82)	--	4.7 (77)

[a]PPM values are average of duplicate analyses, except 11-month delinted seed (single analysis). [b]Concurrent method % recovery; % recoveries are average of duplicate analyses.

Magnitude of the Residue in Plants

Cottonseed. A tolerance of 2 ppm has been established for the residues of N,N-dimethylpiperidinium chloride in or on cottonseed (40 CFR §180.384).

A REFS search dated 9/16/93 listed two BASF Corporation end-use products currently registered for use on cotton, a 0.35 and a 2.01 lb/gal SC/L formulation (EPA Reg. Nos. 7969-52 and 7969-97). Both formulations are registered for foliar applications to cotton using ground or aerial equipment in a minimum of 10 and 3 gal water/A, respectively, with the exception of CA. In CA, minimum application volumes for ground and aerial equipment are 20 and 5 gal water/A, respectively. Ultra low volume (ULV) aerial applications using oil as diluent are also permitted in AL, AR, FL, GA, LA, MO, MS, NC, OK, SC, TN, and TX, in a minimum application volume of 2 pints oil/A. The maximum single application rate is 0.044 lb ai/A and the maximum seasonal rate is 0.066 lb ai/A. A pre-grazing/feeding interval of 30 days and a PHI of 30 days have been established. A grazing/feeding restriction has been established following ULV aerial applications in oil.

BASF Corporation (1993; MRIDs 42734601 and 42734602) submitted data from 57 tests conducted in AL(3), AR(6), AZ(3), CA(6), GA(6), LA(3), MO(3), MS(6), SC(3), TN(3), and TX(15), depicting the residues of mepiquat chloride per se in or on cottonseed harvested 30-33 days following the last of 4, 5, or 6 applications (19 tests each) of the 0.35 lb/gal SC/L formulation. When 4 applications were made, the rates were 0.011, 0.011, 0.022, and 0.022 lb ai/A/application. When 5 applications were made, the rates were 0.011, 0.011, 0.011, 0.011, and 0.022 lb ai/A/application. The four- and five-application tests reflected a total rate of 0.066 lb ai/A, which is 1x the current maximum labeled rate. When 6 applications were made, the rates were 0.011, 0.011, 0.022, 0.022, 0.022, and 0.044 lb

ai/A/application, for a total rate of 0.132 lb ai/A, which is 2x the current maximum labeled rate. The registrant stated that these data were submitted in support of proposed label amendments concerning the maximum number of applications and the maximum application rate. Aerial equipment was used for 21 ULV tests (7 tests per application scheme; MRID 42734601), with spray volumes of 2 pints vegetable oil/A. Ground equipment was used for the remaining 36 tests (12 tests per application scheme; MRID 42734602), with spray volumes of 2-20 gal water/A. The first applications were made 41-87 days after planting, and were repeated at 7-11 day intervals, with intervals of 16-79 days between the next-to-last and the final treatment. Mature cottonseed samples were harvested 30-33 days after the final treatment, and were ginned on the day of collection. Samples were cooled on dry ice or blue ice prior to ginning, and thereafter were stored frozen at ≤ -5 °C for 69-185 days prior to extraction for analysis.

Cottonseed samples were analyzed using the ion chromatography method A9106; the method limit of quantitation was reported to be 0.1 ppm. Residues of mepiquat chloride per se were <0.1-0.76 ppm in or on 38 samples treated at 1x, and were 0.22-1.24 ppm in or on 19 samples treated at 2x (Table 3). Residues were <0.1 ppm (nondetectable) in or on 19 control samples. Concurrent method recoveries were 65-94% from control samples fortified at 0.1, 0.5, 2, and 5 ppm with mepiquat chloride (Table 1 above).

Table 3. Residues of mepiquat chloride per se in or on cottonseed following ULV and multiple low-rate applications, analyzed using method A9106.

MRID	Application Type	No. of Applications	Total Application Rate* (lb ai/A)	Posttreatment Interval (days)	Residues (ppm)	Storage Interval (days)
42734601	ULV (aerial)	4	0.066 (1x)	30	0.12-0.40	74-149
		5	0.066 (1x)		0.10-0.27	
		6	0.132 (2x)		0.23-1.22	
42734602	Multiple low-rate (ground)	4	0.066 (1x)	30-33	<0.10-0.76	69-185
		5	0.066 (1x)		<0.10-0.64	
		6	0.132 (2x)		0.22-1.24	

*The same application rates (lb ai/A/application) were used for ULV and multiple low-rate applications.

Geographic representation is adequate. The test states of AL(3%), AZ(6%), AR(7%), CA(18%), GA(3%), LA(8%), MS(12%), MO(2%), SC(1%), TN(3%), and TX(33%) accounted for approximately 96% of the total 1990 U.S. cotton production (Agricultural Statistics 1991, p. 62). In addition, the ULV test states accounted for 94% of the total 1990 U.S. cotton production in states where end-use product labels allow ULV applications to be made.

The submitted data adequately fulfill outstanding magnitude of residue data requirements for cottonseed, and adequately support the registrant's proposed label amendments, provided that the registrant adequately addresses the concerns raised above regarding extraction of samples for analysis. Residues were ≤ 1.24 ppm in or on cottonseed harvested 30 days following the last of up to 6 applications at 0.011-0.044 lb ai/A/application (total 0.132 lb ai/A; 2x the current maximum labeled rate). These data indicate that residues in or on cottonseed are not likely to exceed the current tolerance of 2 ppm following treatment at the rates tested. Data requirements remain outstanding for cotton forage.

References

Citations for the MRID documents referenced in this review are presented below. Submissions reviewed in this document are indicated by shaded type.

42426801 Burkey, J. (1992) Method for Determination of Mepiquat Chloride Residues in Cottonseed by Ion Chromatography: Lab Project Number: A9106: 92/5064. Unpublished study prepared by BASF Corp. 43 p.

42426802 Burkey, J. (1992) Magnitude of the Residues of Mepiquat Chloride (BAS 083 W), the Active Ingredient in Pix Plant Regulator, in Cottonseed: Lab Project Number: A9128: 92/5015. Unpublished study prepared by BASF Corp. 225 p.

42426803 Burkey, J. (1992) Magnitude of the Residues of Mepiquat Chloride in Processing Fractions of Cottonseed Following Treatment with Pix Plant Growth Regulator: Lab Project Number: A9139: 92/5110. Unpublished study prepared by BASF Corp. 150 p.

42554111 Burkey, J. (1992) Magnitude of the Residue of Mepiquat Chloride and its Metabolites in Cottonseed Raw Agricultural Commodity Samples (1991 Field Program): Lab Project Number: A9212. Unpublished study prepared by BASF Aktiengesellschaft. 115 p.

42734601 Nichols, K. (1993) The Magnitude of Mepiquat Chloride Residues in Cotton Seed Based on Ultra-low Volume Aerial Applications: Lab Project Number: R-92045: 93/5048: 9200537. Unpublished study prepared by Harris Labs and American Agricultural Services, Inc. 177 p.

42734602 Nichols, K. (1993) The Magnitude of Mepiquat Chloride Residues in Cotton Seed Based on Low-rate Multiple Applications: Lab Project Number: A9307: 9200535: R-92044. Unpublished study prepared by Harris Labs and American Agricultural Services, Inc. 200 p.

42892201 Burkey, J. (1993) Freezer Storage Stability of BAS 083 W in Cottonseed and its Processed Commodities: (Mepiquat Chloride): Interim Report: Lab Project Number: 93/5047: 91140: A9305. Unpublished study prepared by BASF Corp., Agricultural Research Center. 54 p.